2019 IMEKO TC-4 International Conference on
Metrology for Archaeology and Cultural Heritage
Florence, Italy, December 4-6, 2019

Measuring instruments and protocols in Archaeomagnetic dating: Magneto-stratigraphy in Archaeology and Volcanology

Principe Claudia[1], Giordano Daniele[1,2], La Felice Sonia[1], Giovannetti Giulio[3], Devidze Marina[4]

[1] *Istituto di Geoscienze e Georisorse, Consiglio Nazionale delle Ricerche, Area della Ricerca di Pisa, Via G. Moruzzi 1, 56124 Pisa, Italy*
[2] *Università degli Studi di Torino, Dipartimento di Scienze della Terra, Via Valperga Caluso 35, 10125 Torino, Italy*
[3] *Istituto di Fisiologia Clinica, Consiglio Nazionale delle Ricerche, Area della Ricerca di Pisa, Via G. Moruzzi 1, 56124 Pisa, Italy*
[4] *M. Nodia Institute of Geophysics, Ivane Javakhishvili Tbilisi State University, Tbilisi, 1 Alexidze str., 0193 Tbilisi, Georgia*

***Abstract* – Information related to the evolution of geomagnetic field in space and time are recorded in the rocks and are the subject of archaeomagnetic studies. These studies allowed us to better understanding the geomagnetic field evolution as well as to improve our comprehension of several geological aspects of the historic evolution of the Earth and human history. Here, based on the description of the functioning principia and models of past and presently used magnetometers we provide, based on our past experience, some suggestions to improve accuracy of archaeomagnetic measurements on fired archaeological materials and volcanic products.**

I - INTRODUCTION

A magnetometer is a magnetic sensor for measuring the direction, strength, or relative change of a magnetic field. The Earth's Magnetic Field (EMF) directional elements are the *declination* (the angle between the horizontal component of the field vector and magnetic north) and the *inclination* (the angle between the field vector and the horizontal surface tangent to the measure point). The third property of the Earth Magnetic field is the intensity (F) that is associated with the length of the magnetic field vector. EMF varies from place to place and in times as due to Earth evolution and planetary interactions.

As revealed to western countries by Marco Polo, the Chinese had discovered the existence of magnets, and the elementary phenomena of magnetism, probably already well over two thousand years before Christ. A detailed description of a compass in China dates back to A.D. 1088 [1]. It is in fact the compass the first kind of magnetometer used to measure the direction of an ambient magnetic field, commonly the Earth's magnetic field. With a magnetic compass, a survey was performed in Sweden in 1640 to detect magnetic iron ores [2]. Although the early discoveries, the eloquent theories of [3] and [4] and the most various applications [5], until the early 40's magnetometers were cumbersome and slow to operate, mechanical instruments that balanced the torque of the magnetic field on a finely balanced compass needle against a restoring force provided by gravity or by the torsion in a suspension (Figure 1). This kind of magnetometers was finally replaced by more sensitive and robust electronic devices (e.g. [6] for a review). Nowadays magnetometers are used both for measuring the EMF parameters or for measuring the Thermo-Remanent Magnetization (TRM) of fired archaeological objects and natural volcanic materials, in order to define their age.

In the first case, and in agreement with their different characteristics, magnetometers are commonly employed in a large number of scientific environments and for various purposes (e.g. geophysical and ore-deposits exploration, climatology, volcanology, biology, archaeology, medicine, agriculture, industry, aerospace, national defence, interdisciplinary research [7-8]).

Archaeological structures or materials heated to high temperatures (tipically volcanic deposits) can acquire, during their cooling a stable TRM which is parallel and proportional to the ambient magnetic field at the time of cooling. The mechanism of TRM acquisition relies on the capacity of some naturally occurring minerals (principally iron oxides) to retain a permanent (i.e.

remanent) magnetization from materials or structures found in situ since the last firing [2].

Here we will revise which are the strengths and weaknesses of magnetometers employed in measuring archaeological findings and volcanic rocks and shortly discuss the possibilities of new upgrades capable of orienting future technological improvements in our laboratory.

II - CLASSIFYING MAGNETOMETERS

The two principal instrumental categories of magnetometers are: (i) vectorial (total field) and (ii) scalar magnetometers. These are defined on the basis of their ability to measure: (i) the magnetic field along the magnetometer sensitivity axis [i.e. a component (vector) of a magnetic field in a particular direction relative to the spatial orientation of the device or its intensity (or strength) value (F)], but not its direction; or (ii) their ability to measure the magnitude of a magnetic field vector (F), using an internal calibration or known physical constants of the magnetic sensor (relative magnetometers). Relative magnetometers measure magnitude or vector magnetic field relative to a fixed but uncalibrated baseline.

According to the physical effects they are based on, the main types of magnetometers can also be classified as it follows:

- Induction magnetometers: the sensors of which are made according to Faraday's electromagnetic induction law.
- Magnetic and magneto-resistive magnetometers: those working by the principle that current in the magnetic field can generate a Lorentz force. For magneto-resistive magnetometers the resistivity of the conductor changes in the magnetic field. This kind of magnetometers can also measure alternating and continuous currents and pulse magnetic fields.

Another important criterion to classify magnetometers is based on the sensitive range and the sensitive resolution of their specific sensors. High sensitivity magnetometers can measure weak field ($< 10^{-7}$ T); medium-sensitivity magnetometers can measure intermediate magnetic field in the range (10^{-7} - 10^{-3} T); low-sensitivity magnetometers are those that can measure large magnetic field ($> 10^{-3}$ T). Low-field sensors (as Superconducting Quantum Interference Devices - SQUIDs) may be capable of detecting fields as low as femtotesla (fT) [9] amongst others).

Major specifications include also the definition of parameters such as: sample rate; bandwidth (or bandpass); noise; resolution; absolute error; thermal stability; sensitivity; gradient tolerance [10, 11].

Actually, the choice of a specified magnetometer strongly depends from the type of survey and from the required analytical results (Figure 2).

Therefore, fluxgate gradiometers are popular due to their compact configuration and relatively low cost. Gradiometers enhance shallow features and negate the need for a base station, while Caesium and Overhauser magnetometers are very effective when used as gradiometers or as single-sensor systems with base stations.

In this short review we will concentrate only on the instruments devoted to the definition of the "vectorial" components (D= declination, I=inclination) of the EMF recorded by fiery objects in the last few millennia. They are essentially four and they will be following described:

1) the Astatic magnetometers of Leopoldo Nobili (1784-1835) is the ancient-most (1825) and now dismissed. It consists of a pair of permanent magnets, of equal moment, mounted antiparallel on a torsion fiber (Figure 1).

Fig. 1. Nobili's astatic galvanometer (Institute and Museum of the History of Science, Florence)

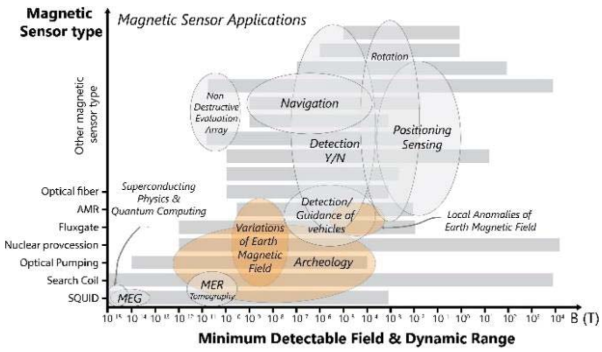

Fig. 2. Magnetic field ranges as associated to specific scientific targets (modified from [12]).

2) The first Induction magnetometer used to search for the Earth's Magnetic Field was the "reversed" magnetometer built by Emile Thellier in 1938, followed by the "Bellevue Magnetometer" built in

the CNRS laboratories of Bellevue in 1956. The Induction magnetometer built in the laboratory of Saint Maur des Fossès by Maxime Le Goff in 1975 and now present in copy also at the Archaeo_Lab laboratories of IGG-CNR in Villa Borbone at Viareggio (LU, Italy) is based on the measurement of the remaining magnetic moments made through two "Helmholtz systems", produced by two identical circular, parallel and coaxial loops, positioned at a distance equal to their radius, that is called Helmholtz’s coil (Figure 3). When the loops are connected in series and are carrying the same current in the same direction, a magnetic field directed along the coil axis is generated, which in the center of the coil can be considered uniform and which brings about the cancellation of the local earth's magnetic field. In the general case, that is, that the radius of the loops is different from the mutual distance h, indicating with D their diameter, the intensity of the field F is expressed by the relation: F = i 10 Π D2n/ (D2/4 + h2/4)3/2 = iC. Where (i) is the intensity of the current and (n) is the number of windings each spiral is made of and (C) is the Helmholtz’s Coil constant. The constructive peculiarities of this magnetometer are described in detail in Thellier's work [13]. This instrument allows very accurate measurement of large samples even in the case of very little magnetization. The disadvantages are the huge dimensions of the samples, the absence of automatization in performing analyses (e.g. rotating the samples) and, most importantly, that this kind of instrument up to now does not exist on the scientific instruments market. All the existing are handmade instruments.

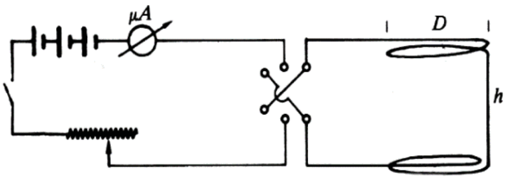

Fig. 3. Simplified scheme of the Helmholtz’s coil

3) The Spinner magnetometer is an Induction magnetometer and belongs to the intermediate sensitivity instruments category. It is constituted by a large sensor coil in which an alternating signal is induced by rotating the sample at high frequency (ca 100 Hz) within the coil. The different components are measured by inserting the sample in the magnetometer each time parallel to one of the three main axes. Spinners are the most widespread magnetometers, largely used in Paleomagnetic studies (see below).
4) The Cryogenic magnetometer is likely the most recent and sensitive instrument for magnetic parameters measurement as its sensor is constituted of a superconducting coil immersed in liquid helium bath at 4 K (at such a low temperature the electric current moves practically without resistance). Being a highly sensitive magnetometer, it is most commonly used to study low-magnetic materials, typically archaeological, but, given its ease to saturate the sensor, it does not offer the possibility to measure, in many cases, volcanic rocks. When a magnetic sample is introduced into the sensor's superconducting area, the magnetic field of the sample creates a current that goes to the superconducting spiral and can then be measured. The advantages of the Cryogenic magnetometer are that it is at least 3-4 orders of magnitude more sensitive than a common Spinner magnetometer, the analysis response is very fast, the sample does not have to be quickly rotated on itself (this allows to measure liquid suspensions, living animals, etc.) and the sample can be automatically rotated in different positions always remaining inside of the sensor area. On the other hand, Cryogenic magnetometer require high level of cooling, making testing expensive and size cumbersome.

III - ANALYTICAL SIMULATIONS OF INDUCTORS FOR ARCHAEOMAGNETIC APPLICATIONS

A. Thellier inductor

This inductor is constituted by a couple of coaxial Helmholtz coils: a principal coil (Rp radius=Dp distance between loops=20 cm, Nc number of loops=6540) and a second coil for magnetic field compensation (Rc radius=Dc distance between loops=40 cm, Np number of loops=1635).
Biot-Savart law was applied for the estimation of the magnetic field distribution of such coil configuration [14], in which the two Helmholtz coils carry opposite electric current I, as shown in Figure 4.

Figure 5a shows the profile plot of the normalized magnetic field pattern in the range 10-30 cm along the z-axis, where the two vertical segments indicate the principal coil loop localizations, while Figure 5b refers to the magnetic field contour plot calculated in the transversal xy-plane to a z-coordinate of 20 mm. It is evident that the magnetic field generated by such coil structure is very homogeneous: such feature is useful for irradiating the sample uniformly in a region where the maximum value of the magnetic field results to be 258 G/A and the maximum variation of magnetic field within a 12x12x12 cm^3 field of view respect to the coil structure centre is 1.22%.

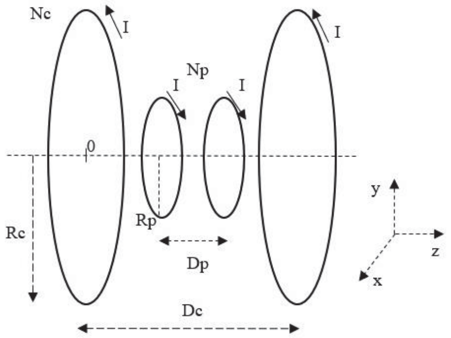

Fig. 4. Coil configuration in Thellier inductor

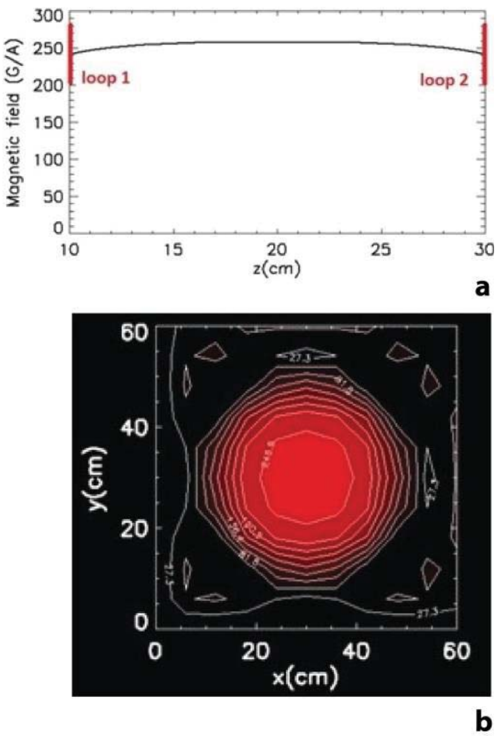

Fig. 5. Magnetic field pattern in Thellier inductor

B. Le Goff inductor

This coil configuration is constituted by a Helmholtz coil as principal coil (Rp radius=Dp distance between loops=20 cm, Np number of loops=10000) and a second coil for magnetic field compensation (Rc radius=40 cm, Dc distance between loops=10 cm, Nc number of loops=2500) carrying opposite electric current I, as shown in Figure 6.

Figure 7a shows the profile plot of the normalized magnetic field pattern in the range 0-20 cm along the z-axis in the space between the two loops of the principal coil, localized as indicated by the two vertical segments, while Figure 7b refers to the magnetic field contour plot calculated in the transversal xy-plane to a z-coordinate of 10 mm. The maximum value of the magnetic field results to be 374 G/A and the maximum variation of magnetic field within a 12x12x12 cm^3 field of view respect to the coil structure center is 0.78%. Such coil configuration provides greater magnetic field intensity and higher field homogeneity respect to Thellier coil.

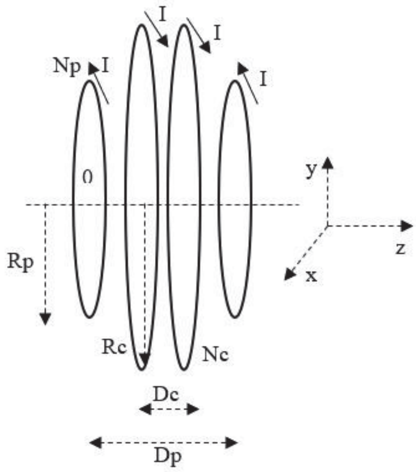

Fig. 6. Coil configuration in Le Goff inductor

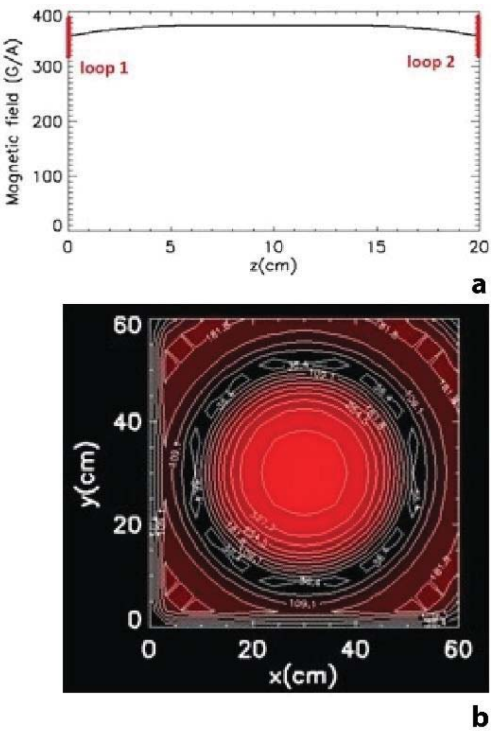

Fig. 7. Magnetic field pattern in Le Goff inductor

IV - ARCHAEOMAGNETIC MEASUREMENTS

Archaeomagnetism finds an early formulation thanks to the intuitions of a restricted number of scientists, many of which of Italian origin. Among these, it is worth emphasizing the contribution of Macedonio Melloni (1798-1854) and Giuseppe Folgheraiter (1856-1913). Both, Melloni and Folgheraiter researches was addressed in considering the magnetic properties of volcanic deposits. Folgheraiter also first applied the study of fossil magnetism to dating ancient pottery, through the reconstruction of the evolution of past Earth's magnetic field [15]. The attribution of an absolute age, through the reconstruction/calibration of the Curves of the Secular Geomagnetic Variation (CSGV), is, actually, one of the more important targets of the nowadays archaeomagnetic studies [16].

Great progress in reconstructing chronologies of baked sediments, archeological features and volcanic rocks sequences has been recently established by various Authors [e.g. 16-19]. Italy in particular, due to its active volcanoes and its particularly rich cultural heritage, is a privileged "playground" to trace back recent variations of the EMF. As a matter of facts, and in addition to the dating activity on furnaces of various age [17], analysis of lava and pyroclastic flows emitted by Italian volcanoes has enabled important steps in understanding and describing geomagnetic directional variations over the past few millennia [e.g. 18, 19]. Archaeomagnetism is a complex discipline, which requires an inter-disciplinary approach frequently involving the knowledge at different levels of both geophysics, statistics, archeology and the ability to correctly interpret the geological (e.g. sedimentary, metamorphic, magmatic and volcanic) environment. The method does not theoretically know extension temporal limits. The stability of the magnetization recorded in the samples, though susceptible to the passage of time, it is generally very strong and can withstand even millions of years.

This fact has led in the past to confuse the two terms archeomagnetism and paleomagnetism in many cases and use them erroneously and indiscriminately. Actually, although based on the same principles of physics, the two methods are applied to substantially different chronological environments and with substantially different analytical instruments and sampling protocols and devices. In particular, archaeomagnetism focus on the analysis of magnetization recorded in rock samples and/or artefacts of archeological origin related to the latter millennia (ca. < 20 ka). On the contrary, paleomagnetic research is carried out by investigating rock samples of geological age. This fact, of apparently negligible methodological importance, implies a different accuracy during sample collection. In fact, in the case of Palaeomagnetic studies, the analyses are focused on finding polarity inversions, or significant (tens of degrees) near-inversions EMF variations; while, in the case of archaeomagnetic investigations a few degrees variations in the Declination and Inclination components of the residual EMF are associated to shifts from centuries to millennia. Therefore, sample collection protocols are of upmost importance in the case of archaeomagnetic studies and many different sample collection strategies are used by different research teams [2]. We will show that in the case of the Large Cell Induction Magnetometers, the preferred sampling method is the Big Sample Plaster Method (BSPM) that, as we will demonstrate, assures a very accurate orientation (with a tenth of a degree accuracy) of the sample. Disadvantage of this protocol is instead the difficulty introduced by the handling and storing of the large ($12x12x12cm^3$) plastered specimens.

V. CONCLUSIVE REMARKS

In order to define their age, TRM of fiery materials are measured using Spinner magnetometers, Cryogenic magnetometers or Large Cell Inductometers. Each one of these instruments had strengths and weaknesses that orient the choice of the analyst. In addition to the problem of building a more automatized inductometer, reduced in dimensions and possible to buy on the market, of upmost importance are both the differences in sensibility of instruments, and the dimensions and shape (cylinders or cubes) of the oriented samples to be inserted in the magnetometer. The ideal sample is perfectly oriented, does not have secondary magnetizations induced by drilling, and is of reasonable dimensions in order to be easily handle and stock. In this sense, the improving of this dating technique is strongly depending both from the instrumental development and from the quality of sampling protocols.

REFERENCES

[1] Lanza R. & Meloni A. The Earth's Magnetism. An Introduction for Geologists. XI, 278 pp, 2006. Berlin, Heidelberg, New York: Springer-Verlag.

[2] Tarling D.D. Archaeomagnetism. In: Encyclopedia of Geomagnetism and Paleomagnetism, Edited by David Gubbins & Emilio Herrero-Bervera. 2007. Edition.

[3] Gauss C. Intensitas Vis Magneticae Terrestris ad Mensuram Absolutam Revocata. Abstracts of the Papers printed in the Philosophical Transactions of the Royal Society of London, 1832, Vol. 3, pp. 166-174.

[4] Maxwell J.C. A Dynamical Theory of the Electromagnetic Field, Philosophical Transactions of the Royal Society of London, 1865, 155, 459–512.

[5] Good A.G. Magnetic world: the historiography of an inherently complex science: geomagnetism in the 20th century. Earth Sciences History, 2007, 26, 281–299.

[6] Mc Henry and Laughlin. Magnetic Properties of Metals and Alloys. In: Physical Metallurgy edited

by David E. Laughlin and Kazuhiro Hono. Elsevier 2015. Chapter 19, pp. 1881-2008.
[7] Mao Z.L. Magnetic Field Measurement. Atomic Energy Press, Beijing, 1985.
[8] Tsao Y.J. & Yang K.C. Technology and Application of Amorphous Alloy sensor, Huahong University of Science and Technology press, Wuhan, 1998.
[9] Rikpa P. Magnetic sensor and magnetometers. Artech House Remote Sensing Library, 2001.
[10] Lowrie W. Fundamentals of Geophysics Cambridge University Press, Cambridge. 5th ed. 2004.
[11] Magnetometer Technology (Chapter 9). In Micro and Nano Technologies, Space Microsystems and Micro/nano Satellites. Editor: Zheng You, Pages 341-360, 2018. Butterworth & Heinemann.
[12] Díaz-Michelena M. Small Magnetic Sensors for Space Applications. Sensors 2009, 9, 2271-2288.
[13] Thellier E. Sur la direction du champ magnétique terrestre, en France, durant les deux dernières millénaires Phys. Earth Planet. Int., 24 1981, pp. 89-132.
[14] Giovannetti G., F. Frijia, A. Flori, D. Montanaro, Design and simulation of a Helmholtz coil for Magnetic Resonance Imaging and Spectroscopy experiments with a 3T MR clinical scanner. Applied Magnetic Resonance, 2019, 50 (9), 1083-1097.
[15] Folgheraiter G (1899). Sur les variations séculaires de l'inclinaison magnétique dans l'antiquité. J. Phys., 8, pp. 660-667
[16] Pavón-Carrasco, F. J., Osete López, M. L., Torta, J. M., De Santis, A. A geomagnetic field model for the Holocene based on archaeomagnetic and lava flow data. Earth Planet. Sci. Lett., 2014, 388, 98–109.
[17] Tema, E. Archaeomagnetic Research in Italy: Recent achievements and future perspectives. In: *The Earth's Magnetic Interior,* IAGA Special Sopron Book Series, 2011, Vol. 1, Ch. 15, pp. 213-233. Eds: Petrovsky, E., Herrero-Bervera, E., Harinarayana, T., Ivers, D., Springer.
[18] Tanguy JC, Le Goff M, Chillemi V, Paliotti A, Principe C, La Delfa S, Patanè G. Secular variations of the geomagnetic field direction recorded in lavas from Etna and Vesuvius during the last two millennia. C R Acad Sci Paris Earth & Planetary Sciences, 1999, 329: 557–564
[19] Tanguy, J.C., Le Goff, M., Principe, C., Arrighi, S., Chillemi, V., Paiotti, A., La Delfa, S., Patanè, G. Archaeomagnetic dating of Mediterranean volcanics of the last 2100 years: validity and limits. Earth Planet. Sci. Lett., 2003, 211, 111–124.